Labyrinth structures in Zircaloy-4 as a consequence of strain ageing effects on fatigue

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Dislocation structures in Zircaloy-4 samples fatigued at 623, 713 and 823 K were studied by transmission electron microscopy techniques. The samples, cycled with a total strain rate $2 \times 10^{-3}\ s^{-1}$, show a linear cyclic hardening rate which is maximum at temperatures where stronger strain ageing effects were reported. A two-wall structure similar to the labyrinth structure observed in f c c metals was found. This structure is attributed to the enhanced dislocation activity produced by strain ageing phenomena.

1. Introduction

The bulk of the microstructural observations on fatigued samples have been performed on single- and polycrystals of metals which possess a face centred cubic (f c c) crystal structure. It is known that for not too high a strain amplitude, and depending on the stacking fault energy, planar arrays of dislocations and persistent slip bands (PSB) are the dominant dislocation features in such metals [1]. Metals of f c c structure with a PSB dislocation structure show, as well as veins and ladders, a two-dimensional wall structure which has been called "labyrinth" [2].

Most of the studies found in the literature on labyrinth structure are related to its configuration, and not on to the apparition conditions. Since such a structure almost always appears accompanying PSBs, and knowing that the formation of these appears to be triggered at a critical density of dislocations [3], one can infer that the labyrinth structures in a given material are more likely to occur in conditions with an enhanced dislocation activity, as for example during strain ageing.

There is, to our knowledge, only one work in the literature where the existence of PSBs in hexagonal, closed, packed (h c p) metals is reported. Kwadjo and Brown [4], working with magnesium single crystals, have shown that the properties of the PSBs are quite similar to those of PSBs in f c c metals. They did not find any labyrinth structure in this material. They also observed that the hardening curves of this h c p metal show initial rapid hardening followed by stages of low but non-zero hardening rate. In comparison with the hardening behaviour observed in f c c metals, where a saturation stage is usually found after the rapid hardening producing the PSBs, this could be a distinctive characteristic for hexagonal metals.

The present work attempts to propose that for h c p materials a critical dislocation density is also needed to produce PSBs as labyrinth structures. This critical dislocation density will be obtained if the dislocation formation mechanisms are able to produce an enhanced dislocation activity. This enhanced activity may be produced by locking dislocations such as occur during strain ageing phenomena. Strain ageing effects during fatigue have been reported on this material [5]. Between 673 and 773 K, Zircaloy-4 samples became stronger during the cycle. This effect was thought to be a consequence of an enhanced work hardening rate produced by dislocation–solute–atom interactions. The higher work-hardening rates during strain ageing have been generally shown to be associated with increased rates of dislocation accumulations [6].

This note presents the first results of transmission electron microscope (TEM) investigations on Zircaloy-4 samples to decide whether a labyrinth structure is also developed in h c p metals, and to study the influence of strain ageing effects on the formation of this structure.

2. Experimental procedures

From longitudinal rods of Zircaloy-4, prepared in accordance with ASTM B550 Grade 704, hour-glass-shaped cylindrical specimens were machined (for detailed testing conditions and the survey of specimens, see [5]). Strain cyclic tests were made under total axial control using a triangular elongation–time function. The total strain range used for most of the test was $\Delta\varepsilon = 0.01$ and the total strain rate $\dot{\varepsilon} = 2 \times 10^{-3} s^{-1}$. The tests were performed in the temperature range 623–823 K where strain ageing effects were reported

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[5]. Discs for TEM examinations were sectioned both in perpendicular (transverse) and parallel (longitudinal) directions to the specimen axis, i.e. the applied stress. After conventional preparation techniques, the thin foils were investigated in a Philips EM 300 electron microscope with an acceleration voltage of 100 kV.

3. Results

3.1. Mechanical behaviour

Fig. 1 shows the stress response of Zircaloy-4 for strain-controlled cycling for three temperatures: 623, 713 and 823 K. At first sight one could infer a very different hardening behaviour between these temperatures. However, on plotting the number of cycles in a linear scale, a common feature of the curves is evident. Three stages can be observed on each of the curves. Stage I is characterized by a high but decreasing hardening rate; stage II starts when stage I becomes almost constant. In this stage the peak tensile stress, σ, varied linearly with the number of cycles for the examined temperatures. A least-squares regression analysis of the experimental results of samples fatigued at 713 K, where a maximum in hardening rate was observed, gives

$$\sigma = 160.2 + 0.05\,N \quad (\sigma \text{ in MPa}) \tag{1}$$

This analytical expression, where N is the number of cycles, is followed between 10 and up to 1000 cycles for this temperature and these mechanical conditions. After a maximum of the peak tensile stress, stage III begins which shows softening caused by the failure.

Tests performed at 713 K with a different total strain range but the same total strain rate show similar mechanical behaviour (Fig. 2). The three curves present the same hardening rates and the maximum in the peak tensile stress remains almost the same.

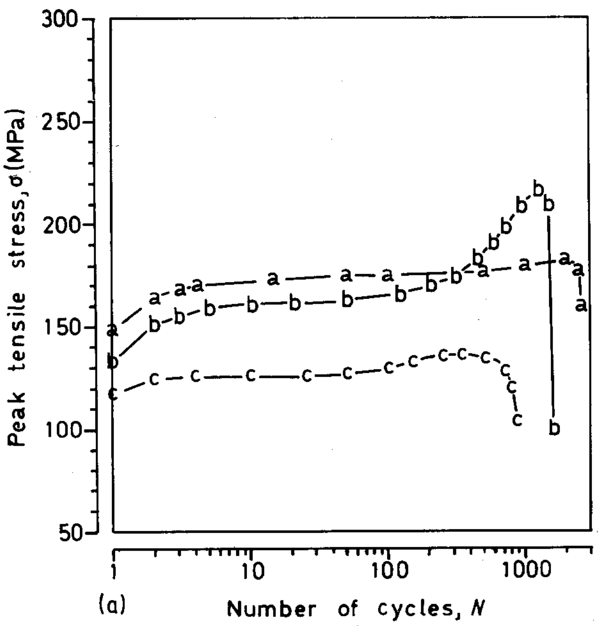

Figure 1 Peak stress response as a function of the number of cycles ($\Delta\varepsilon$ = 0.01, $\dot{\varepsilon} = 2 \times 10^{-3}\,s^{-1}$). (a) 623; (b) 713; (c) 823 K.

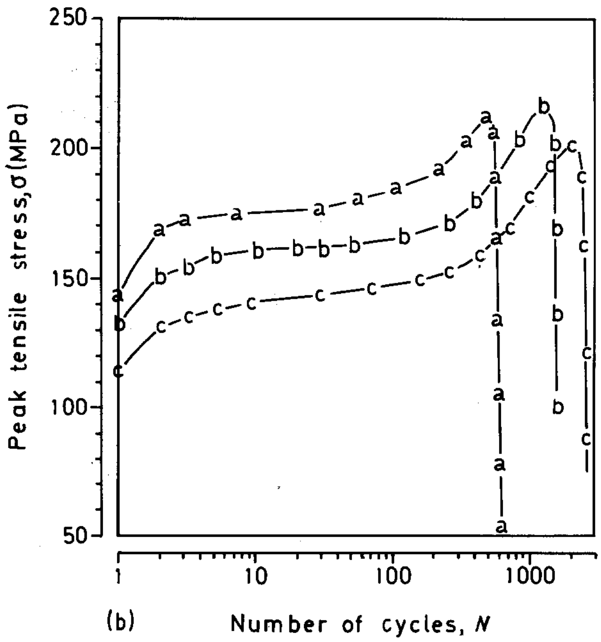

Figure 2 The cyclic stress response of Zircaloy-4 at 713 K and $\dot{\varepsilon} = 2 \times 10^{-3}\,s^{-1}$.

3.2. Microstructural observations

The deformation substructures of samples cycled up to fracture at different temperature values, but with the same mechanical conditions, are shown in Figs 3–5.

The most prominent dislocation structure observed at 623 K, corresponding to a minimum cyclic hardening rate of the linear region, is a vein-like structure (see Fig. 3). The non-uniformity of the dislocation distribution is remarkable, and regions of high-(veins) and low-(channels) density of dislocations occupied similar volume fractions. The veins consisted mainly of primary edge dislocations and the channels, approximately 0.4 μm wide, of screw dislocations.

At 713 K, corresponding to the curve with a maximum peak tensile stress and a linear hardening rate with the cycles, dislocation walls in two directions were the dominating feature (Fig. 4). The walls were of two types: those perpendicular to the primary slip

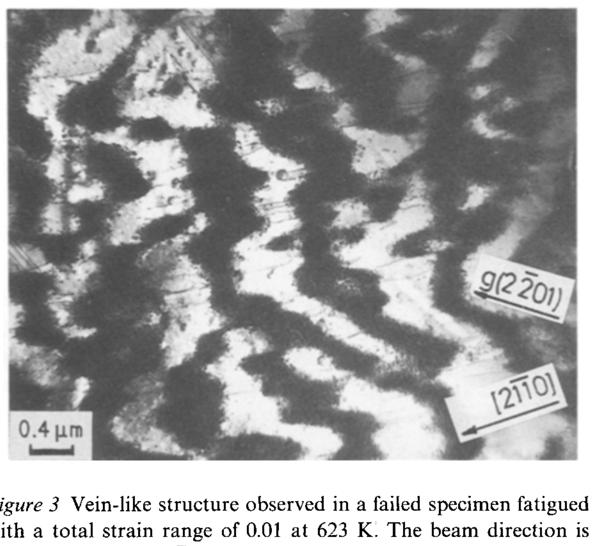

Figure 3 Vein-like structure observed in a failed specimen fatigued with a total strain range of 0.01 at 623 K. The beam direction is $\bar{B} = [\bar{1}2\bar{1}6]$; $\bar{g} = (2\bar{2}01)$.

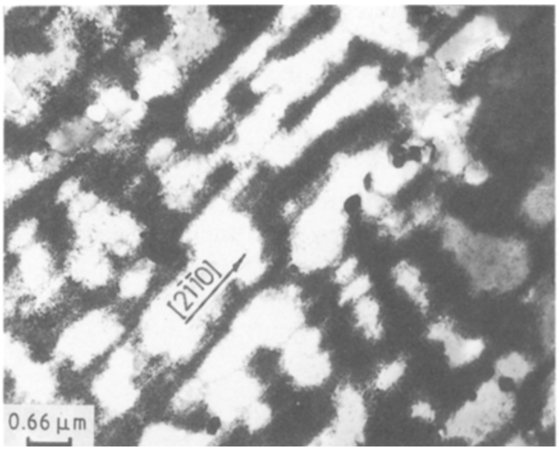

Figure 4 Wall structure observed in a failed specimen fatigued with a total strain range of 0.01 at 713 K. The beam direction is $\bar{B} = [2\,\bar{1}\,\bar{1}\,0]$.

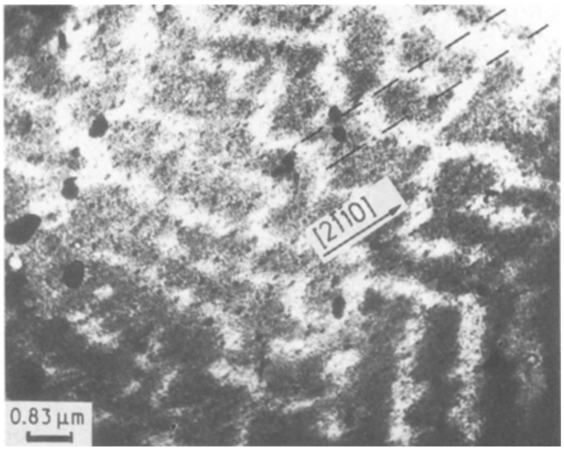

Figure 6 Labyrinth-like structure observed in a failed specimen fatigued with a total strain range of 0.0075 at 713 K. The beam direction is $\bar{B} = [\bar{1}\,2\,\bar{1}\,6]$; $\bar{g} = [1\,\bar{2}\,1\,1)$.

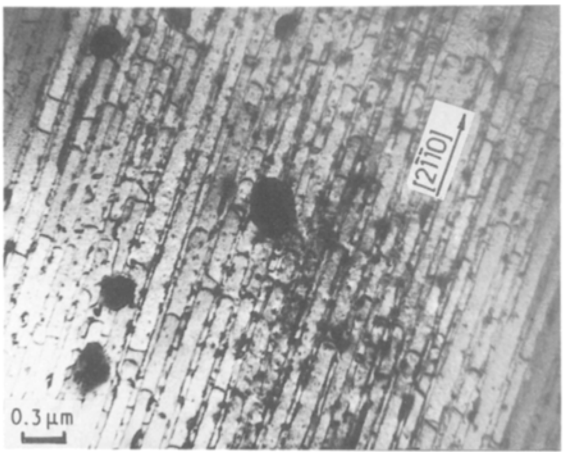

Figure 5 Well developed band structure observed in a failed specimen fatigued with a total strain range of 0.01 at 823 K. The beam direction is $\bar{B} = [0\,\bar{1}\,1\,2]$; $\bar{g} = (2\,\bar{1}\,\bar{1}\,0)$.

direction consisted of primary edge dislocation bundles, and those parallel to this one could be built by dislocations of the $\langle 1\,1\,\bar{2}\,3\rangle$ systems. This aseveration comes from an analysis with the invisibility criterion applied to (0 0 0 2) reflections.

At 823 K, a completely different dislocation structure was found (Fig. 5). The dislocations were arranged in a band structure showing dense walls of primary screw dislocations with a scarce density of edge dislocations inside the channels linking the walls. This structure was previously reported by the authors [7].

Fig. 6 corresponds to a specimen cycled up to fracture with a total strain range $\Delta\varepsilon = 0.0075$ and a total strain rate $\dot{\varepsilon} = 2 \times 10^{-3}\,s^{-1}$ at 713 K. The dominating features were dislocation walls mainly formed in a structure very similar to the labyrinth pattern observed in f c c metals. The two sets of walls which compose the labyrinth structure do not belong to defined crystallographic planes, but they lay in directions approximately parallel to the traces of two $\{0\,\bar{1}\,1\,2\}$ planes. In this micrograph is also indicated the $[2\,\bar{1}\,\bar{1}\,0]$ crystallographic direction, and it can be observed that this direction limits short dislocation walls that could be equivalent to the 'ladders' in the classical PSB ladder-like structure of f c c metals.

4. Discussion

The cyclic hardening rate observed for the different temperatures could be attributed to an increased rate of dislocation accumulations as product of dislocation–solute–atom interactions. It is known that this type of interaction, which is strongly temperature dependent, could produce an important drag on the dislocation movement. At lower temperatures, between room temperature and 623 K, this type of interaction is not so strong, the cyclic hardening resulting from the mutual trapping of primary edge dislocations. The trapped dislocations accumulate in bundles oriented perpendicular to the direction of the primary Burger's vector. The bundles, in turn, act as obstacles to further dislocation motion and progressively harden the specimen. Since the strain amplitude is not high enough, only dislocations veins are finally formed from the original bundles [8]. This structure remains approximately the same during cycling and up to failure.

From Fig. 1, it is evident that the strain ageing phenomena observed at 713 and 823 K have the strongest influence on samples cycled at 713 K. Although the cyclic hardening rate is almost the same for both temperatures, these phenomena actuate during more time at 713 K due to the higher number of cycles up to failure occurring at this temperature. Stronger interactions between solute atoms and dislocations could originate an increased amount of dislocation storage. The stronger interactions come from the enhanced solute diffusion consequence of vacancies produced during fatigue deformation. This dislocation accumulation will produce an enhanced dislocation activity which would be responsible for an increase in dislocation density and premature activation of secondary slip systems. It is known that the activation of more than one active slip system will originate a cell structure [9]. Only cell structures were observed at

713 K, cycled with a total strain range $\Delta\varepsilon = 1\%$. As a consequence of cell formation, the material becomes harder and the cell formation rate would be related to Equation 1.

The increased dislocation density resulting from the locking of mobile dislocations by solute atoms can give rise (when the samples are not cycled at high enough amplitudes to produce cells or the premature failure of the sample) to a two-wall structure that could be seen as equivalent to the labyrinth structure observed in fcc metals (Fig. 6). In this figure are also marked regions of the specimen which may be considered as the embryos of a PSB ladder structure. This term only refers to $\{1\,0\,\bar{1}\,0\}$ walls of dislocations arranged regularly along a direction parallel to the trace of the basal plane. Whether the existence of this structure is related to similar maxima found in the peak tensile stress for curves cycled with different total strain range (Fig. 2) remains unknown.

In Fig. 5, a high-temperature structure similar to that reported in [7] is observed. At 823 K, high stresses which could be produced by an enhanced dislocation density as a consequence of strain ageing are relieved by climb and cross slip processes. These two mechanisms are helped by the enhanced rate of vacancy production which occurs in fatigue testing.

5. Conclusions

The present study on hcp Zircaloy-4 samples deformed cyclically in a temperature range where strain ageing effects are present has led to the following conclusions.

1. The stress increases linearly with the number of cycles as a consequence of the enhanced dislocation activity produced by dislocation locking.

2. At temperatures where the strain ageing effects are present for longer, a two-wall structure similar to the labyrinth structure observed in fcc metals was found.

Acknowledgements

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